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The Step From Laboratory to Industry

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Of the 120 million bushels of apples we produce annually, about one-fifth are culls or surplus. The problem arises as to what should be done to keep them from going to waste.

First, a chemist is assigned the job of developing new products. He familiarizes himself with the chemical characteristics of the apple and then with the actual operations in the various apple-processing plants. From that study, he gets an idea that he develops into a process for making sirup from the juice. The method consists of treating apple juice with lime and heating to 180° F., clarifying, adding acid, and evaporating it to 75 percent solids. The sirup looks good to him, because it is mild in taste, sweet, of good color, and easy to make.

Now that he has a new product, many questions occur to him. What use can be made of the sirup? If it is to be used for food, would it require better apples than if it is to have an industrial use? What would be the difference in price? How much will it cost to make the sirup commercially? What price can he get for the sirup? How much can be sold? Who would make it? Who would use it? Is it worth while to investigate it in the pilot plant?

At that point, the liaison man enters the investigation. In industry, he may be known as a technical salesman, market researcher, industrial liaison specialist, fact finder, or sales serviceman. In the Department of Agriculture, he is all of those; he bridges the gap between the laboratory and industry. His responsibility is to make sure the maximum value is obtained from the research accomplishment by developing to the greatest possible degree its utilization by industry. One way to describe the bridging process is to continue with the actual example of apple sirup. The description of its commercial development, which begins now, and that of essence recovery, which follows, illustrate the liaison man's procedure.

To reach an intelligent decision on the desirability of pilot-plant operation, more information was needed. As a starting point, the liaison man got from the chemist samples of the sirup and a pamphlet entitled *Production of Bland Apple Sirup from Apples*, which describes the method of preparation, the physical and chemical characteristics of the sirup, and suggestions as to uses.

Then, the liaison man discussed the development with trade associations, publishers of trade magazines, farmers, cooperatives, and representatives of industry. The preliminary inquiries showed enough commercial interest in the manufacture and use of the sirup to justify experimental studies in the pilot plant.

While the liaison man awaited results from the studies, he assembled crop statistics which would be helpful in selecting the best places for plants. The statistics included the quantities of apples available in different areas, percentages of culls and wastes, seasonal availability, prices, and the amount processed for various uses.

In preparation for the commercial tests on the sirup to determine its possible application, he found out the kind and amount of sweeteners and hygroscopic (moisture-holding) substances used by the different industries, because those were the main characteristics of apple sirup. Cane sugar was taken as an example of a sweetener and glycerin of a hygroscopic material.

When substantial quantities of sirup became available from the pilot plant, the liaison man arranged with representative industrial concerns for actual performance tests. He closely followed the tests to discover any problems that might require solution by the laboratory.

As a sweetener, apple sirup was evaluated in beverages, ice cream, candy, drugs, bakery products, and fountain and table sirups, as a modifier of curd tension in milk, and as a source of carbohydrates for infant feeding. It was also tested as a hygroscopic agent and plasticizer—or softener—in cosmetics, tooth paste, cork closures for bottle caps, coated paper, bakery goods, and tobacco. The tobacco industry normally uses 40 million pounds of glycerin a year.

Although all the tests gave promising results, some difficulties made further investigation in the laboratory necessary. A few examples show their nature. When apple sirup was used as a sweetener, especially in table sirups and beverages, a bitter aftertaste developed. The sirup did not mix satisfactorily when used in drugs, cosmetics, and tooth pastes that contain oils and fats. Spray residues from apples used for sirups made for human consumption could be a hazard. The liaison man brought the problems of commercial application to the laboratory for solution. The chemists traced the bitter aftertaste and poor mixing to the relatively high calcium content of the sirup. They remedied the trouble and reduced the spray residue within safe limits by special treatment—ion exchange.

In the instances in which he learned the sirup could be used successfully, the liaison man obtained more complete information, such as a comparison of its performance and price with those of other materials, the potential demand in different industries, advantages and disadvantages, possible modifications in the sirup or method of use, and the industries in the best position to manufacture or use it successfully.

Upon completion of the commercial information and engineering data on plant design, process details, operating constants, yields, and costs from the semiplant-scale studies, a revision of the apple sirup publication was in order. Equipped with the new publication, *Bland Apple Sirup,* which covered all phases of the development, and a supply of the sirup, the liaison man directed efforts to the creation of the largest possible industrial utilization of the process. That he did by disseminating information on the development and by helping commercial concerns in the solution of other problems in connection with large-scale production or use. If it seemed likely that the problems would hamper the utilization of the apple sirup, he referred them to the Government laboratory.

In that stage of the introduction of the development to industry, he found that prospective manufacturers generally were unwilling to undertake the expense of new equipment unless they were assured of a substantial demand for the sirup. The prospective users did not want to assume the cost of thorough application tests unless they were certain of a reliable source of supply. Consequently, commercial interest had to be stimulated in the production as well as utilization of the sirup.

As a result, production got into full swing in 15 plants. Some manufacturers built plants exclusively to make sirup. Others, like plants for evaporating milk and maple sirup, had idle equipment during the fall apple season, since spring was their normal busy period.

The market demand for sirup developed until it exceeded 4 million pounds annually. Most of it was used by the cigarette industry to retain moisture in tobacco. Two manufacturers installed special equipment—ion exchangers—to produce sirup of reduced calcium content for use primarily in cosmetics, drugs, tooth paste, beverages, and table sirup. A large dairy-products company recently developed a modified apple sirup to compete in the 12-million-dollar market of sirups for infant feeding.

After the Second World War, the demand for apple sirup dropped sharply, because more sugar and glycerin were available for civilian uses. The sirup could not compete with glycerin, because it was less efficient as a hygroscopic agent and had a color and taste that were undesirable for certain applications. It could not compete with sugar because of its color, taste, and much higher cost. As a table sirup, it was too expensive, and its flavor was too weak to mix with the cheaper sugar sirups. Urgent requests were received from industry to find a way to remove color and taste. But because any additional processing would result in an even higher price in comparison with competitive sweeteners, evidently the best solution was to remove it from such competition by enhancing its natural flavor characteristics. A laboratory development, apple essence, proved to be the solution. It greatly improved the flavor of apple sirup.

APPLE ESSENCE was the result of several years of investigation by laboratory chemists and engineers to find a method for preparing a full-flavor, concentrated apple juice, which would make a beverage on dilution with water, with all the characteristics of fresh apple juice. H. P. Milleville, a chemical engineer, made the goal possible when he invented a method for recovering and concentrating, unaltered, the volatile flavors, or essence, normally lost during the evaporation of the juice of fresh apples.

To obtain commercial opinion, the liaison man called on representative manufacturers of table and fountain sirups with samples of essence-flavored apple sirup. He was told that the sirup was too expensive to compete successfully with the cheaper sugar sirups an too weak for use as a flavor in the sirups. Blending the essence with the concentrated juice from tarter apples finally produced a satisfactory flavor material. The blend made a delicious apple drink when it was diluted with water. Mixed with sugar sirup, it made a good table sirup.

The discussion with industrial concerns disclosed that, although interest was great in essence-flavored table sirup and the full-flavor concentrate used in beverages, far more important was the value of the essence itself as a flavor material for many products. The engineers prepared a publication that emphasized the method for the recovery of the essence rather than the preparation of apple-flavor table sirup or concentrate.

To stimulate the production of essence, the liaison man called on manufacturers of apple sirup, concentrated apple juice, vinegar, and brandy, because they were in a good position to use the essence-stripped juice.

With increased production, the price of essence dropped from 15 dollars to 5 dollars a gallon. The market expanded.

The beverage industry became interested in the essence for use with either the concentrated apple juice or sugar-base sirup, to which they planned to add acid and coloring matter. The inclusion of the concentrate, although preferable from the standpoint of naturalness of flavor and nutritional value and as an outlet for the residual juice from the recovery process, was unsatisfactory because of increased costs and poor stability. Stability problems, the most difficult, were the formation of a cloudy precipitate, loss of flavor, and development of a mold growth. Improvement in methods of clarification or the use of sterilizers corrected the cloud formation. The acid apple-juice concentrate apparently caused the loss of flavor, as the essence was stable when it was stored separately. Beverages that contained fruit-juice concentrates spoiled more quickly than those with efined sugar sirups because sugar sirups do not support so well the growth of micro-organisms as do fruit juices that are high in minerals and nitrogenous substances.

Because the conventional heat sterilization would result in loss of volatile essence, the makers of beverages that contained concentrated juice had to resort to germproof (Seitz) filtration, the addition of preservatives, or the removal of mineral and other nutrients with ion-exchange treatment to check spoilage. Eventually, most of the essence went into beverages.

Many candy manufacturers thought the essence would be too expensive and volatile for large-scale use. The technicians, by a study of the application of essence to many types of candy, found a method for making a pectin-gel type that soon went into commercial production.

Several manufacturers of dairy products reported successful results in the development of essence-flavored milk beverages, ices, sherbets, and ice cream. Later they noted a loss of essence flavor in milk and ice-cream products. They attributed the loss to the absorption of the flavor by the milk fats. Makers of ice cream overcame the difficulty by adding the essence to the ribbon of ice or fruit that is included in some types of ice cream. The industry, impressed by the possibilities of the different fruit essences as flavoring, requested that they be included in standards of identity for ice cream.

Tobacco manufacturers also reported that the essence greatly improved the flavor of a number of products. But, because of its rapid disappearance, essence would be of no commercial value to them unless a method could be found to prevent its loss.

The liaison man referred the problem of retaining the essence flavor in commercial products to the laboratory and flavor specialists in industry. The criticisms and suggestions he received from manufacturers of flavor extracts were especially helpful in making suggestions for improving the essence.

Several industrial concerns pointed out the marked variations they had noted in the strength and quality of different apple essences, concentrates, and mixtures. Some of the concentrates quickly developed a disagreeable odor and taste. Methods were needed to insure absolute standardization and uniformity.

The laboratory studied the influence of apple varieties by separately recovering the essence and concentrating the essence-stripped juice from nine varieties. Marked differences were noted in the quality of the essences and the stability of the concentrates when they were evaluated individually and in beverages, jelly, and candy. The blending of selected varieties seemed to be the solution. The chemists, however, did not discover any satisfactory tests to measure the strength of the essence, because volatile flavors are complicated and intangible. Consequently, the strength continues to be expressed in terms of the relative quantity of juice from which it was recovered. For example, 150-fold essence means 1 gallon of essence recovered from 150 gallons of juice.

A problem arose because of the apparent loss or change of flavor in different commercial products to which the essence had been added. A flavor specialist pointed out the necessity of making an exact determination of the constituents of apple essence before it was possible to develop satisfactory procedures for stabilizing the flavor in different commercial products. A chemist, Jonathan W. White, Jr., was assigned the task. After many months of work, he was able to identify 26 compounds—no small accomplishment, for the compounds represent only about 0.5 percent of the 150-fold essence (50 parts per million of the original apple juice). This information gave industry a basis for studies to find suitable methods for stabilizing essence in different commercial products.

The makers of flavoring extracts directed attention to the fact that the commercial demand for apple flavor was much less than the demand for grape, strawberry, raspberry, peach, cherry, orange, and pineapple flavors. They asked us to develop suitable procedures for recovering the essence from the other fruits. We started studies on grapes, strawberries, and oranges. Commercial manufacturers began to produce grape, peach, raspberry, orange, pineapple, and strawberry essences, some of which have been produced so far on only an experimental scale. Approximately 50 essence units, with capacities up to 5 million gallons of juice a year, were installed.

THE SERIOUS PROBLEM as to the economical utilization of the residual juice after the essence had been recovered was referred to the laboratory. The flashed juice of apples could be used for apple sirup, concentrated apple juice, and such, if there was a market for such products. However, the demand for the sirup was seriously curtailed; the demand for the concentrated juice was limited mainly to the preserve industry. A study was needed of ways to expand present markets for the products. It was not feasible to express the juice of the other fruits for essence recovery. As a result, the laboratory and industry initiated investigations to develop satisfactory procedures for recovering the essence from crushed fruits and the condensate from preserve manufacture.

A request for help was received from a manufacturer who had been informed that the apple, grape, peach, and cherry essences, which he had imported from Canada, were subject to an Internal Revenue tax of 9 dollars a gallon. Analyses of the samples showed that their alcohol content ranged from 1 to 8 percent. Essences containing 0.5 percent or more were subject to the tax. Laboratory engineers who worked on the problem devised a new method for making an essence with only one-third the normal alcohol content—they vaporized only 3 percent, instead of the usual 10 percent, for subsequent concentration.

Several companies complained th the imported essences gave a bitter taste and contributed practically no flavor. They traced the trouble to the citric acid denaturant in the essences they used. Exempt from tax were imported essences that contained a denaturant of 6 ounces of citric or tartaric acid to a gallon or 25 percent of sugar.

A COMMERCIAL DEMAND developed for essences of higher concentration, because they would contribute greater flavor value for the same tax cost and would be useful for flavoring certain dried products. To meet the problem, the engineers worked out modifications in the equipment and process which made possible the production of 800-fold essence.

A later law exempted essence from the Internal Revenue alcohol tax if several requirements were met.

Manufacturers of flavoring extracts directed our attention to the desirability of developing equipment and processes that would combine the best features of the conventional alcohol flavor-extract method and the essence process.

Processors questioned the use of the term "essence" for the product, as implied an alcoholic extract. They suggested that the phrase "volatile fruit concentrate" would be better, even though it is longer.

SO THE ACCOUNT is brought up to date, as of spring, 1951. It brings out two points: How the gap is bridged between laboratory and industry, and how the laboratory men are alert to help solve the problems of production and utilization that may arise when industry takes the product of the test tube into the factory.

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